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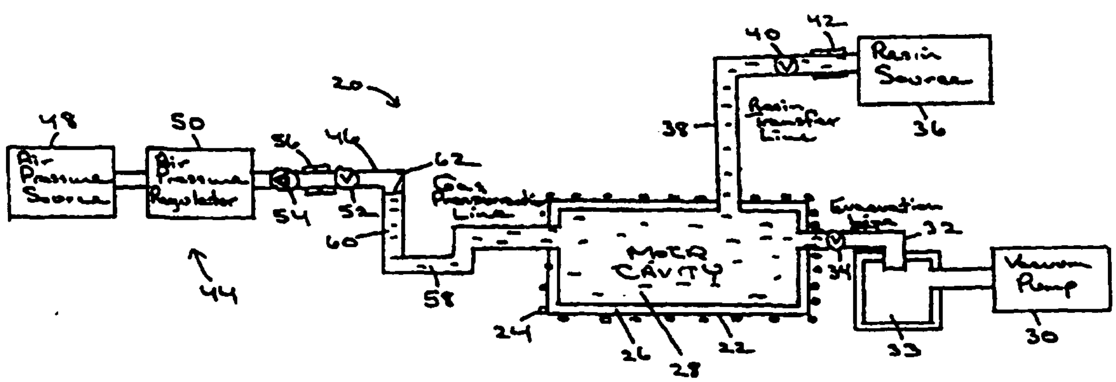

(57) Abstract

Composite articles are prepared by resin transfer molding and an applied gas pressure during heating. A mass of reinforcement is placed into a mold (22), and the mold (22) is evacuated. A resin source (36) is connected to the mold (22), and thermosetting resin is introduced into the mold (22) from the source (36). A gas pressure source (44) is connected to the mold (22), and an external column (40) of resin is maintained in the gas pressure line (46). The mold (22) is heated to set and cure the resin within the mold (22), but the resin in the external column (40) is maintained in an unset condition. After the resin is introduced into the mold (22) and simultaneously with the heating of the resin, a gas pressure is applied to the column (40) of liquid resin, preferably at a pressure greater than the vapor pressure of water at the temperature to which the resin is heated, to prevent gas evolution to form bubbles during setting and curing and to minimize the volume of any bubbles already present in the resin from other sources.

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METHOD OF RESIN TRANSFER MOLDING

BACKGROUND OF THE INVENTION

This invention relates to the manufacture of composite material articles, and, more particularly, to the fabrication of such articles by resin transfer molding.

A composite material is a material made from two or more starting materials, in which the physical identities of the starting components are retained in the final product but the properties of the final product are superior to those obtained when the components are used separately. An example of a composite material finding widespread industrial application is carbon or other types of reinforcement fibers embedded in a matrix of a polymeric resin such as an epoxy. The fibers impart a directionally controllable high strength and/or stiffness to the final composite material product. The matrix protects the fibers, imparts fracture toughness to the composite material, and permits structural use and attachment of the material.

Composite materials can often be fabricated precisely to their final shape and size, minimizing post-fabrication machining and other working operations. Various techniques have been developed for fabricating articles of composite materials. One such approach is the fabrication of composite articles by resin transfer molding (RTM). In resin transfer molding, the fibers or other reinforcement are placed into a mold in the desired arrangement. The mold is evacuated, and a liquid thermosetting resin is introduced into the mold through a resin transfer line from a resin source under a slight pressure. The mold is heated (or was initially preheated) to cause the resin in the mold to set. The resin is thereafter cured by maintaining the elevated temperature for a sufficient period of time.

Gas present in the resin prior to curing and trapped in place by the curing operation can adversely influence the properties of the final composite material. Such gas can be introduced into the resin from external sources, or

it can evolve from the resin itself. As an example of the former, air bubbles may be initially present in the liquid resin or may be mixed into the liquid resin as it flows into the mold. As an example of the latter, during the curing operation dissolved water vapor can evolve from the resin to form bubbles in the resin, if no pressure is applied to the resin.

In either case, the bubbles of gas typically cannot find their way out of the curing resin matrix, because at this stage the resin has set and cured to a solid form so that the bubbles are trapped. It is therefore conventional practice to maintain the external pressure of the resin source using the resin introduction apparatus or to valve off the resin transfer line to capture the expansion pressure. The resin matrix material expands against this back pressure during heating and curing, suppressing water vapor bubble formation and minimizing the volume of entrapped air.

The inventor has discovered that this approach to bubble suppression, while operable, has several drawbacks. The bubble suppression procedure is largely uncontrolled, as it is determined in large part by the expansion and curing characteristics of the resin, not any process control determination. The expansion of the resin is normally much greater than that of the mold, so that resin extrudes between the mold halves. As pressure is relieved in this manner, gas can leak into the mold and form gas pockets in the composite material that are detrimental to its final physical and mechanical properties. Also, if the pressure is maintained by the introduction apparatus, the apparatus is unavailable for other operations for the entire period of curing the resin, which may require several hours. These problems arise during the curing stage of the processing rather than the introduction and setting stages.

There is therefore a need for an improved approach to the processing of composite material articles produced by resin transfer molding. The present invention fulfills this need, and further provides related advantages.

SUMMARY OF THE INVENTION

The present invention provides an approach to the fabrication of composite-material articles having a thermosetting resin matrix by resin transfer

molding. Bubble formation in the matrix and the composite article is avoided, leading to a sound final product with optimal physical and mechanical properties. Process control is improved, as the technique used for bubble suppression during processing is more readily controlled than are the prior procedures. The approach of the invention also improves the productivity of the process, because it permits the resin introduction apparatus to be disconnected and removed from the mold immediately after introduction of the liquid resin, for use with other molding operations. Additional apparatus is required for the practice of the present approach, but that apparatus is minimal and inexpensive.

In accordance with the invention, a method for the resin transfer molding of reinforced composite articles comprises the steps of providing a mold and placing a reinforcement mass into the mold. A resin source is provided in communication with the interior of the mold, and thermosetting resin is introduced into the mold from the resin source. The resin in the mold is heated to cause the resin to set and cure. After the introduction of resin into the mold is complete and simultaneously with at least a portion of the step of heating, a gas pressure is applied to the resin.

The gas overpressure is preferably applied by maintaining a column of liquid resin in a gas pressurization line that extends from the gas pressure source to the mold, and applying gas pressure to that column of liquid resin. No pressurization gas actually contacts the resin within the mold using this approach. Air can therefore be used as the pressurization gas. The gas pressurization line is designed with the upper surface of the liquid resin in a vertical section of the resin transfer line, so that the pressurizing gas is above the liquid resin. Bubbles of the pressurizing gas cannot work past the liquid resin, and no foreign substance contacts the curing resin within the mold. Additionally, the use of a gas pressurization line separate from the resin transfer line permits the resin introduction apparatus to be disconnected and used elsewhere during heating of the mold, while maintaining the gas pressure on the resin in the mold.

This approach provides flexibility in the fabrication approach. The gas overpressure can be readily controlled to meet the requirements of particular

types of resins, curing schedules, and mold designs. In a typical application, where dissolved water vapor in the resin is otherwise the principal cause of bubbles in the final product, the gas overpressure is controlled to be greater than the vapor pressure of water at the temperature reached during the concurrent portion of the step of heating. The gas overpressure can be varied as necessary during the heating process.

The application of gas pressure in this manner is to be clearly distinguished from its application during the introduction of the resin into the mold. It is well known to apply a gas pressure as the resin is introduced into the mold, to cause the liquid resin to flow into the mold. Steps are typically taken during resin introduction to minimize the presence of gas, such as degassing the resin and evacuating the mold. By contrast, in the present case the gas pressure is applied after introduction of the resin into the mold and continued during at least a portion of the time that the resin is heated, at least until the resin begins to set.

The present approach is not used to advantage with thermoplastic resins and other types of molding operations. Thermoplastic resins are injected in their molten state and quickly harden upon cooling, rather than injected relatively cool and hardened upon heating as for thermosetting resins. They have relatively low gas evolution during setting and curing. And, as a practical matter, a thermoplastic resin would harden in the present apparatus and prevent the subsequent application of gas pressure.

The present approach provides an advance in the art of the fabrication of composite articles by resin transfer molding. Sound articles with minimal bubbles or voids are produced, with improved productivity as compared with prior approaches. Other features and advantages of the present invention will be apparent from the following more detailed description of the preferred embodiment, taken in conjunction with the accompanying drawings, which illustrate, by way of example, the principles of the invention.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic view of an apparatus used to prepare composite

materials by the present approach; and

Figure 2 is a process flow block diagram for the preferred approach to practicing the present invention.

DETAILED DESCRIPTION OF THE INVENTION

Figure 1 depicts an apparatus 20 for the fabrication of articles by resin transfer molding, according to the present invention. The article is prepared in a mold 22 that can be controllably heated by a heater, here shown schematically in the form of heating coils 24. The mold 22 has walls 26 that define a mold cavity 28 of the desired shape and size. The interior of the mold 22, the mold cavity 28 can be controllably evacuated by a vacuum pump 30 that pumps the mold cavity 28 through a vacuum line 32, liquid isolator 33, and vacuum valve 34.

Liquid resin is transferred to the mold cavity 28 from a resin source 36 through a resin transfer line 38. The resin source is connected to the resin transfer line 38 through a valve 40, and is also connectable and disconnectable at a coupling 42. The resin transfer line 38 has a trap section 44 that includes a vertically extending section 46, between the resin source 36 and the mold 22.

Gas pressure is applied to the resin within the mold cavity 28 from a gas pressure source 44 through a gas pressurization line 46. The gas pressure source 44 and the gas pressurization line 46 are preferably separate from, and independent of, the resin source 36 and the resin transfer line 38. The gas pressure source 44 includes, in the preferred embodiment, an air pressure source 48 and an air pressure regulator 50. The gas pressurization line 46 includes a valve 52 to close the line 46 and a check valve 54 to prevent resin from flowing from the mold cavity 28 into the gas pressure source 44. The gas pressurization line 46 is connectable and disconnectable to the mold 22 at a coupling 56. The gas pressurization line 46 has a U-shaped trap section 58 that includes a vertically extending section 60 that defines a column of resin, between the gas pressure source 44 and the mold 22.

Figure 2 depicts a preferred method for using the apparatus 20 of Figure 1 to accomplish resin transfer molding of articles. The mold 22, which is

typically a split-mold design, is provided, numeral 70. With the mold 22 open, reinforcement fiber and/or other reinforcement material is placed into the mold cavity 28, numeral 72. The reinforcement is furnished and arranged in the manner required in the finished part. The reinforcement can be, for example, unidirectional fibers of a single type arranged uniformly throughout the mold cavity, or a mixture of fiber types, or fibers positioned with intentionally nonuniform arrangements. Mats or three dimensional arrays of fibers can also be used. The present invention is operable with any known type, arrangement, and volume fraction of the reinforcement, which can be maintained in place when resin is transferred into the mold. After the reinforcement is arranged in the mold cavity 28, the mold 22 is closed and sealed.

The resin source 36 is provided, numeral 74, along with the resin transfer line 38 and other components described previously.

With the valves 40 and 52 closed, the mold cavity 28 is evacuated by opening the valve 34 and operating the vacuum pump 30, numeral 76. The mold is typically evacuated to a pressure of about 1/4 atmosphere or less, to remove gas that could otherwise be incorporated into the final composite.

After the evacuation is complete, the valve 34 is closed, and the valve 40 is opened. Liquid thermosetting resin is transferred from the resin source 36, through the resin transfer line 38, and into the mold cavity 28, numeral 78. The resin is typically forced through the resin transfer line 38 under a slight pressure to induce fluid flow. The liquid resin fills the mold 22 and at least that portion of the gas pressurization line 46 reaching into the vertically extending section 60. At this point, the mold cavity 28 contains a mixture of solid reinforcement and liquid resin.

Various types of liquid thermosetting resins can be used. The resin must have a sufficiently low viscosity as a liquid to be transferrable through the resin transfer line 38, with viscosities typically in the range of 10-20,000 centipoise, and most preferably less than about 4000 centipoise at the introduction temperature. A wide range of thermosetting resins are available for resin transfer molding, including, for example, epoxies, bismaleimides, and cyanate esters. There is no known limitation on the type of thermosetting resin with which the present invention is operable, except as discussed herein. As

discussed previously, thermoplastic resins are not used in the resin transfer molding operation.

The resin source 36 may optionally be disconnected, numeral 80, at any time after the resin is introduced and the valve 40 is closed, by disconnecting the coupling 42. (Although step 80 is illustrated in Figure 2 as preceding steps 82 and 84, it could be accomplished concurrently with these steps or omitted entirely.) The use of a separate gas pressure source 44 to apply pressure to the resin in the mold cavity 28 permits the resin source 36 to be (optionally) disconnected and used elsewhere during the heating cycle, which may last for several hours. Similarly, the vacuum pump 30 can be (optionally) disconnected for use elsewhere, at any time after the completion of evacuation step 76 and the closing of the valve 34, by disconnecting its coupling. The resin source 36 and/or the vacuum pump 30 can thereby be used in a more economical fashion than possible for the approach where both remain connected during the entire subsequent heating operation.

By its nature, the resin typically contains liquids or gasses that are present from the introduction step 78 or are evolved during the subsequent heating step. For example, the resin may have air bubbles or dissolved water that turns to water vapor when the resin is heated. If the resin has already set to a solid or semi-solid state, the bubbles of air, evolved water vapor, or other gas cannot escape from the interior of the solid mass. The gas bubbles within the solid are therefore retained to the final product, and their presence adversely affects the physical and mechanical properties of the final product.

The formation of gas bubbles during curing has been known in the past, and a typical approach to minimizing the problem has been to apply a fluid pressure to the resin transfer line and thence to the resin within the mold cavity, or to close the valve 40 during heating so that the natural expansion of the resin creates a high internal pressure within the mold. This back pressure, if greater than the vapor pressure of water or other dissolved gas in the resin at the curing temperature, suppresses the formation of bubbles. These approaches are not fully satisfactory, however, because of several reasons. If a pressurized resin source is used, the resin source must remain connected to the mold throughout most or all of the entire curing cycle, which may last many hours.

If the valve 40 is simply closed to permit a back pressure to develop, the back pressure may cause the resin to extrude from the seams of the mold. In the latter case, gas can flow back into the mold cavity through the extrusion opening and into the article, causing further incorporation of gas bubbles.

In the presently preferred approach, the separate gas pressure source 44 is provided, and gas pressure is applied to the resin in the mold cavity 28 through the medium of the resin column present within the gas pressurization line 46, numeral 82. The trap section 58 of the gas pressurization line 46 is preferably filled with resin so that a resin/air interface 62 lies at the top of the resin column within the vertically extending section 60. The position of the resin/air interface 62 is adjusted to lie above the level of the resin in that portion of the gas pressurization line 46 between the interface 62 and the mold cavity 28. The gas pressure from the gas pressure source 44 is applied to the resin column at the resin/air interface 62, and this gas pressure is thence imparted to the resin within the mold cavity 28. Applying a pressure to the resin within the mold cavity 28 in this manner ensures not only that the gas supplying the pressure does not contact the resin in the mold directly, but also that an air bubble cannot enter the resin within the mold cavity 28. This positioning of the interface 62 is best accomplished by making the trap section 58 of the resin transfer line 46 out of transparent or translucent material such as polytetrafluroroethylene (teflon) tubing, and introducing sufficient resin to place the interface 62 within the vertically extending section 60, with the valve 52 closed. The valve 52 is thereafter opened to place the gas pressure source 48 in communication with the surface of the resin. The check valve 54 is set to block any back flow of resin that might be unintentionally passed through the valve 52, to prevent damage to the regulator 50.

The resin within the gas pressurization line 46 remains liquid during the entire procedure, as it is not heated to the setting or curing temperatures by the heating coils 24. If any resin within the gas pressurization line 46 does set, that portion of the line 46 is cut out of the line and discarded, and that section of the line is later replaced. Gas pressure applied to the surface of the resin, at the resin/air interface 62, is transmitted through the resin in the line 46 to the mold cavity 28, and thence to the curing mass of resin within the mold cavity

28. The pressure is desirably maintained at a sufficiently high level to suppress the evolution of dissolved gas at the curing temperature, so that no bubbles can form, but not at such a high pressure that the mold could be damaged. The preferred air pressure is from about 60 to about 120 psig (pounds per square inch, gauge).

In the situation of most practical concern, the evolution of water vapor from water dissolved in the resin, to form water vapor bubbles, is suppressed by maintaining the applied gas pressure above the vapor pressure of water at the temperature reached during the step of heating. The vapor pressure varies as a function of temperature, and accordingly will vary with the preferred curing temperature for various types of resins. For a typical curing temperature of about 350°F, the vapor pressure of water is about 60 pounds per square inch. The applied gas pressure from the gas pressure source is maintained above that value during heating.

In other situations, different potentially evolving gases may be of greater concern. An important virtue of the present approach is that the gas pressure can be controlled in any required manner so that gas evolution into the setting and curing solid resin can be entirely suppressed or partially suppressed if some internal porosity is desired. The size of porosity, when present, can also be controlled by varying the applied gas pressure. Various pressurization/time schedules can be followed, depending upon the process requirements. That is, it may be determined that the most beneficial pressurization schedule requires a variation in pressure (not just maintaining a constant high pressure) during the heating step 84, and the present approach allows such a schedule to be readily followed by varying the output pressure of the air pressure regulator 52 with time.

The mold 22 is heated, numeral 84, to a temperature at which the thermosetting resin in the mold sets, using the heating coils 24. The mold 22 may be partially preheated to this temperature before the resin transfer, particularly if the setting requires a sufficiently long period of time. Thus, the step 84 may not necessarily be accomplished completely after the step 78, and may to some extent be concurrent with the introduction step 78. The setting temperature, setting time, curing temperature, curing time, and other details of

the heating procedure are specific to each type of resin, and will be known to those in the art of resin transfer molding. As an example, however, for epoxy resin, setting and curing may be accomplished at a temperature of about 350°F in a time of about 60 minutes.

Thermosetting resins exhibit several stages in progressing from the fully liquid state of the introduction step to their final cured state. Setting of the resin from a liquid to a solid or semi-solid form occurs first. The resin is thereafter cured to its hardened state. After curing, the article is at least sufficiently strong for removal from the mold and handling. Post-curing operations to reach even higher strength levels can be conducted for some resin types after the article has been removed from the mold, in a separate furnace-treating operation.

The temperatures and times employed in the heating step 84 are determined by the type of resin employed. The mold 22 may be heated to a single temperature for a period of time to accomplish both setting and curing. Alternatively, the mold 22 may be heated to a first, lower temperature for a first period of time to accomplish setting, and thereafter heated to a second, higher temperature for a second period of time to accomplish curing.

In any case, the gas pressure application 82 is commenced after the introducing step 78 is complete. The gas pressure application 82 and heating 84 proceed concurrently for at least a portion of the heating step 84 until the resin has set. After the resin is set, the internal structure in respect to bubble presence, position, and size is largely established and does not substantially change during subsequent curing, in most cases. For such situations, the gas pressurization step 82 can be discontinued after the resin has set. In other cases, it may be desirable to continue the gas pressurization until the resin is partially cured. The demarcation between setting and curing of the resin within the mold cavity 28 is not exact in most cases, particularly because the temperature within the mold cavity 28 can vary with location and because the distinction between the end of setting and the beginning of curing is sometimes not easy to identify. Because maintaining the moderate gas pressure into the curing regime usually does not have an adverse effect upon the final product, it is therefore usually desirable to continue the gas pressurization step 82 until

the resin is well into its curing phase.

After the curing step 84 is complete, the power to the heating coils 24 is gradually reduced in the manner prescribed for curing the selected resin. After the temperature is cooled sufficiently, the mold 22 is opened. The article within the mold, a composite material having reinforcement embedded within a matrix of the cured resin material, is removed for use or, if appropriate, further processing such as post-curing.

Although a particular embodiment of the invention has been described in detail for purposes of illustration, various modifications and enhancements may be made without departing from the spirit and scope of the invention. Accordingly, the invention is not to be limited except as by the appended claims.

CLAIMS

What is claimed is:

1. A method for the resin transfer molding of reinforced composite articles, comprising the steps of:

providing a mold;

placing a reinforcement mass into the mold;

providing a resin source in communication with the interior of the mold;

introducing thermosetting resin into the mold from the resin source;

heating the resin in the mold to cause the resin to set and cure; and, after the step of introducing and concurrent with at least a portion of the step of heating,

applying a gas pressure to the resin.

2. The method of claim 1, wherein the step of applying a gas pressure includes the step of

applying an air pressure.

3. The method of claim 1, wherein the step of applying a gas pressure includes the step of

applying a gas pressure that is greater than the vapor pressure of water at a temperature reached during the step of heating.

4. The method of claim 1, wherein the step of applying a gas pressure includes the step of

applying a gas pressure of from about 60 to about 120 pounds per square inch-gauge.

5. The method of claim 1, including the additional step, before the step of introducing, of

evacuating the interior of the mold.

6. The method of claim 1, wherein the step of providing a resin

source includes the step of

connecting the resin source to the mold through a resin transfer line, and wherein the step of applying a gas pressure includes the steps of

supplying a gas pressure source, and

connecting the gas pressure source to the mold through a gas pressurization line, the gas pressurization line being separate from the resin transfer line.

7. The method of claim 1, including the additional step, after the step of introducing and before completion of the step of heating

disconnecting the resin source from the column of resin and connecting a source of gas pressure to the column of resin.

8. The method of claim 1, wherein the step of providing a resin source includes the step of

connecting the resin source to the mold through a resin transfer line, and wherein the step of applying a gas pressure includes the step of

supplying a gas pressure source, and

connecting the gas pressure source to the mold through a gas pressurization line, the gas pressurization line being separate from the resin transfer line.

9. The method of claim 1, wherein the step of introducing the thermosetting resin includes the step of

introducing thermosetting resin into the mold from the resin source, leaving a column of resin external to the mold;

wherein the step of heating the resin includes the step of

heating the resin within the mold to cause the resin to set and cure, the resin in the column external to the mold being maintained unheated, unset, and uncured;

wherein the step of applying a gas pressure includes the step of

applying a gas pressure to the column of unset resin after the step of introducing and concurrently with at least a portion of the step of heating, such that gas cannot leak into the mold, the gas pressure being greater than the vapor

pressure of water at the temperature reached during the concurrent portion of the step of heating; and

including an additional step, after the step of introducing and before completion of the step of heating, of

disconnecting the resin source from the column of resin while continuing the step of applying a gas pressure.

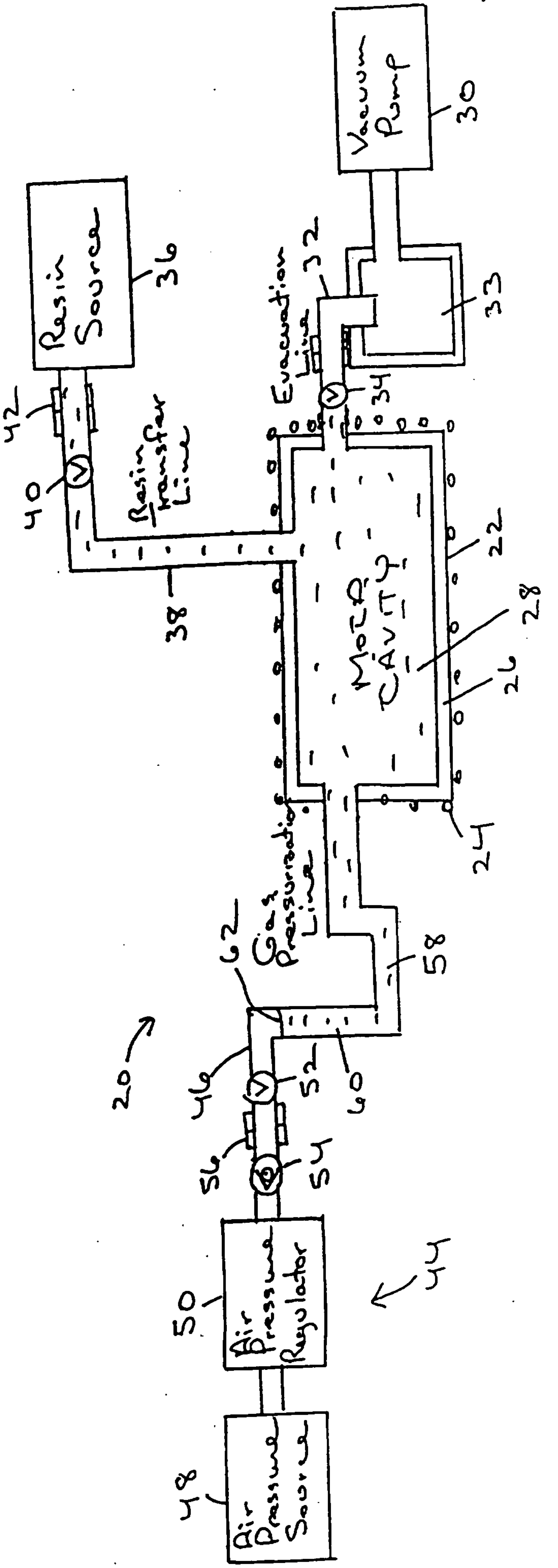
Vacuum Pump
30
Resin Source
36
Evacuation Line
32
33
34
42
40
Resin transfer Line
38
MOLD CAVITY
22
28
26
24
Gas Pressurization Line
62
58
20
46
52
60
56
54
44
50
Air Pressure Regulator
48
Air Pressure Source

Fig 1

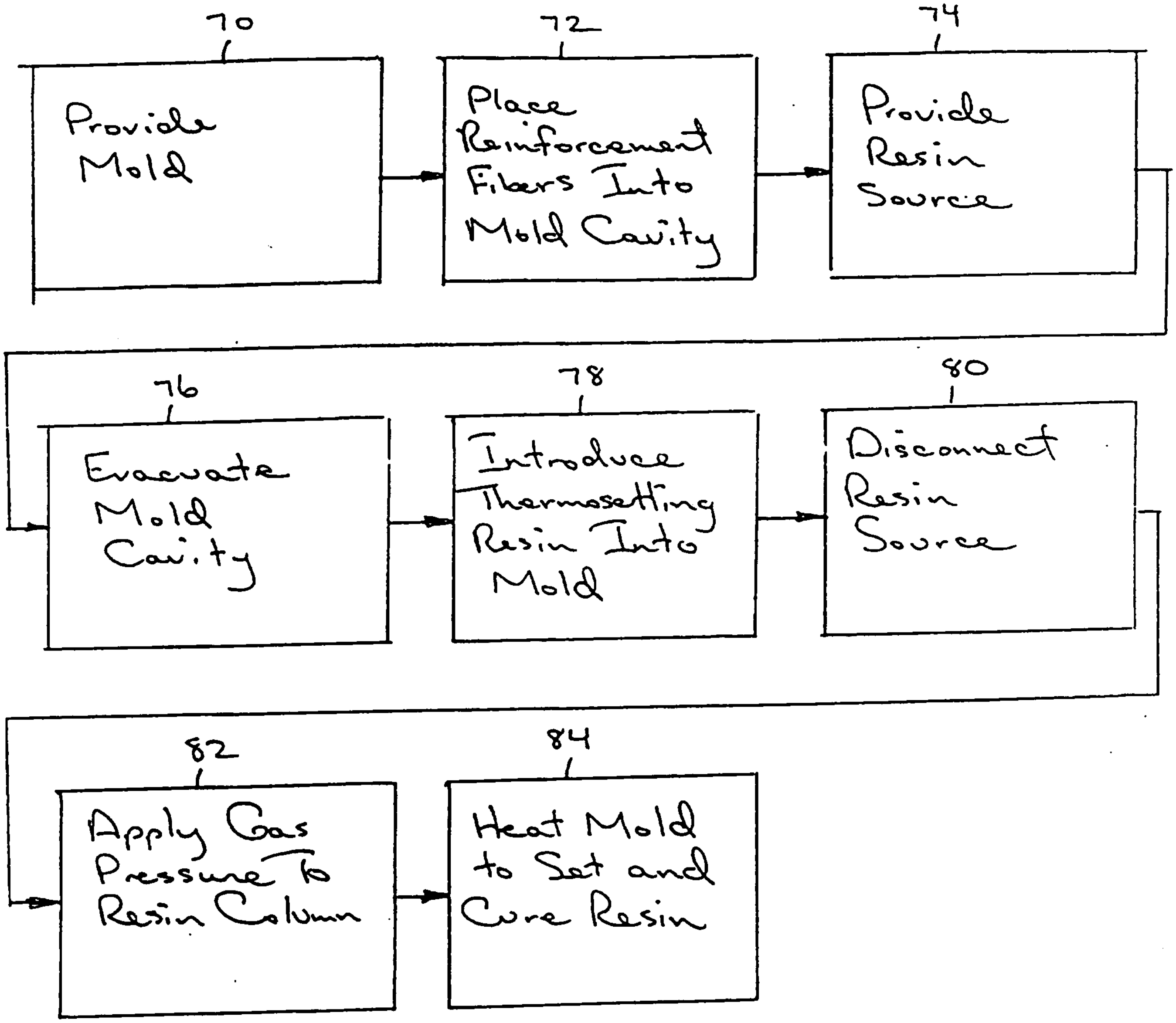
70
Provide Mold
72
Place Reinforcement Fibers Into Mold Cavity
74
Provide Resin Source
76
Evacuate Mold Cavity
78
Introduce Thermosetting Resin Into Mold
80
Disconnect Resin Source
82
Apply Gas Pressure To Resin Column
84
Heat Mold to Set and Cure Resin

Fig 2

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US96/00719

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :B29C 45/00, 47/00; B29D 9/00
US CL :264/510, 513, 102, 328.1, 328.4, 328.9, 328.16
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 264/510, 513, 102, 328.1, 328.4, 328.9, 328.16

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 2,435,610 (SCHNEIDER) 10 FEBRUARY 1948, ENTIRE DOCUMENT.	1-9
Y	US, A, 3,044,118 (BERNHARDT ET AL) 17 JULY 1962, ENTIRE DOCUMENT.	1-9
Y	US, A, 3,670,066 (VALYI) 13 JUNE 1972, ENTIRE DOCUMENT.	1-9
Y	US, A, 4,563,324 (KUBAT ET AL) 07 JANUARY 1986, ENTIRE DOCUMENT.	1-9
Y	US, A, 4,810,444 (ALBERINO ET AL) 07 MARCH 1989, ENTIRE DOCUMENT.	1-9
Y	US, A, 4,923,666 (YAMAZAKI ET AL) 08 MAY 1990, ENTIRE DOCUMENT.	1-9

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
23 APRIL 1996	20 MAY 1996

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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